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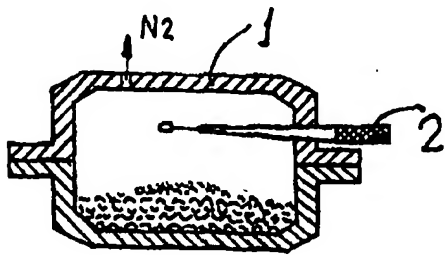
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<p>(21) International Application Number: PCT/IE99/00144 (22) International Filing Date: 23 December 1999 (23.12.99) (30) Priority Data: 981100 23 December 1998 (23.12.98) IE (71) Applicant (for all designated States except US): KINGSPAN RESEARCH AND DEVELOPMENTS LIMITED [IE/IE]; Dublin Road, Kingscourt, County Cavan (IE). (72) Inventors; and (75) Inventors/Applicants (for US only): MURTAGH, Adrian [IE/IE]; 4 Hillcrest, Tullinadaly Road, Tuam, County Galway (IE). FEERICK, Patrick, Joseph [IE/IE]; Emeracly, Milltown, Tuam, County Galway (IE). (74) Agents: O'BRIEN, John, A. et al.; John A O'Brien & Associates, Duncairn House, 3rd floor, 14 Carysfort Avenue, Blackrock, County Dublin (IE).</p>		<p>(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p>Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</p>
<p>(54) Title: ROTATIONAL MOULDING METHOD AND APPARATUS</p> <p>(57) Abstract</p> <p>A method of rotation moulding comprises forming a reactant thermoplastic melt in a mould (1, 20). The reactant thermoplastic melt comprises a mixture of thermoplastic pre-polymer, such as lactam (12) and a polymerisation activator. The pre-polymer and activator may be mixed in the mould under an inert atmosphere or a premix of the pre-polymer and activator may be formed prior to delivery into a mould (1). The premix may be stored under an inert atmosphere in a sealed container such as a bag (16) from which it may be delivered into a mould (20) at room temperature. Alternatively the reactant melt may be formed in the mould (1) by delivering a pre-polymer into the mould and injecting a polymerisation activator into the mould.</p> 		

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ROTATIONAL MOULDING METHOD APPARATUSIntroduction

5 The invention relates to rotational moulding.

Rotomoulding is an inexpensive process that is widely used for manufacturing hollow plastics components. A polymer resin in a powder form is introduced into a hollow mould which is heated externally in a large oven. The mould is biaxially
10 rotated as the powder sinters and the plastic coats the interior surface of the mould relatively evenly, typically to a thickness greater than 3 mm. Such conventional rotomoulding has the advantages of relatively low capital equipment costs, inexpensive tooling as there is no pressure applied, and allows large volume complex shapes to be easily moulded.

15 There are however, a number of serious disadvantages with conventional rotomoulding. These include general lack of proper control of the moulding process which leads to product imperfections and possible post-moulding warpage because no pressure is applied. Therefore, conventional rotomoulding technology
20 is generally used for low cost large scale components.

Various thermoplastic materials have become available which have potential advantages of improved mechanical strength, toughness, low water absorption, high softening temperature and relatively low cost.

25 There is a need for an improved rotomoulding process that will convert some of the recent developments into a commercially viable process and apparatus.

This invention is directed towards providing such an improved process and
30 apparatus.

Statements of Invention

According to the invention there is provided a method of rotomoulding comprising the step of forming a reactant thermoplastics melt in the mould.

5

In one embodiment of the invention the reactant thermoplastics melt comprises a mixture of a thermoplastic pre-polymer and a polymerisation activator.

10

The thermoplastics pre-polymer and the polymerisation activator may be mixed in the mould. Alternatively the thermoplastics pre-polymer and the polymerisation activator are mixed prior to delivery into a mould.

Most preferably the mixing and preferably also the moulding is carried out under an inert atmosphere.

15

The inert atmosphere is typically a nitrogen atmosphere.

20

In a preferred embodiment the pre-polymer and polymerisation activator are mixed to form a pre-mix which is stored in a holding container prior to delivery into the mould.

Preferably the mixture is added to the mould at ambient temperature conditions.

Preferably the mixture is introduced into the mould under an inert atmosphere.

25

In a preferred embodiment after introduction of the mixture, the mould is heated to polymerisation temperature. Preferably, after introduction of the mixture, the mould is rotated, usually biaxially, typically at a speed of greater than 5 r.p.m.

30

In another embodiment of the invention the method comprises the steps of: -

- 3 -

delivering a thermoplastics pre-polymer into the mould; and

introducing a polymerisation activator to the thermoplastic pre-polymer in the mould.

5

In this case the polymerisation activator may be introduced into the mould separately from the pre-polymer. The polymerisation activator may be injected into the mould to form a reactant melt in the mould.

10

In a preferred embodiment of this aspect the method includes the steps of:

placing the thermoplastic pre-polymer in a mould;

heating the mould;

15

injecting an activator into the pre-heated material in the mould; and

further heating the mould.

20

In one aspect the method comprises the steps of: -

placing thermoplastic pre-polymer in a mould;

heating the mould;

25

rotating the mould;

injecting an activator into the preheated material in the mould; and

30

further rotation and heating of the mould.

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Preferably in this case the mould is pre-heated to a temperature of from ambient temperature to 170°C, preferably to a temperature of approximately 170°C.

5 In one embodiment the mould, after injection of the activator, is further heated to a temperature of from 170°C to 205°C over a period of from 5 to 10 minutes. Preferably the mould is further heated to a temperature of about 200°C over a period of about 10 minutes to ensure complete polymerisation.

10 In a preferred embodiment the activator line is purged after injection of the activator.

Preferably the method includes the step of rotating the mould during preheating.

15 In one embodiment the method includes the step of rotating the mould after injection of the activator to dissolve or disperse the activator in the preheated melt.

20 The mould is preferably rotated biaxially after dispersion of the activator in the melt. The biaxial rotation is usually at a speed of greater than 5 rpm.

In one preferred embodiment of the invention the thermoplastic is selected from one or more of PBT, ABS, acrylic, polycarbonate, lactam, mixtures, blends or copolymers thereof.

25 In one case the thermoplastic is a lactam material. In this case the activator is preferably an activator for producing an ionically polymerised lactam.

Preferably the lactam is lactam-12.

30 The invention also provides a rotomoulding apparatus including a mould having an injector means for injecting an activator into the mould for in-mould reactive

- 5 -

moulding. In this case preferably the apparatus includes a flush means for flushing the injector after injection of the activator into the mould.

5 Preferably the mould used in the method of the invention has integral mould heating and cooling means.

The rotation speeds during rotomoulding may be changed from an initially high speed when the liquid has a low viscosity to lower speeds as the viscosity of the matrix within the mould increases.

10 Nitrogen flush is maintained throughout to ensure polymerisation and to reduce oxidation and material degradation.

15 The invention further provides a rotomoulded article whenever produced using the apparatus and/or method of the invention.

Brief Description of Drawings

20 The invention will be more clearly understood from the following description thereof given by way of example only with reference to the accompanying drawings, in which: -

25 Figs 1 to 5 are schematic views illustrating the various steps in a process According to one embodiment of the invention; and

Figs 6 to 11 are schematic views illustrating steps in a process according to another embodiment of the invention.

30

Detailed Description

The method and apparatus for rotomoulding used in the invention employs a system with improved control of the mould temperature. This facilitates a high level of repeatability and traceability. A gimbal-type aluminium framed biaxial rotomoulding machine may be used to provide full biaxial rotation. The mould may be heated by infra-red. Alternatively a composite tool with embedded heating wires and cooling channels may be used. One such system is described in WO-A-961496.

Referring in particular to Figs 1 to 5 in the method according to one embodiment of the invention a reactant thermoplastics melt is formed in the mould rather than outside the mould. This has processing advantages especially in overcoming difficulties in handling the separate monomeric and activator materials. In this case they are kept separate and are only mixed in the mould so that in mould polymerisation of the material takes place. This is easy to control because the material can be kept in motion in the mould and the temperature is readily controlled in the mould. Thus, an even and reproducible polymerisation and moulding can be achieved.

Granules/pellets of lactam-12 are first placed in a mould 1 at room temperature. The mould 1 is then pre-heated to approximately 170°C to melt the lactam-12. The mould is preferably rotated during pre-heating to maximise exposure of pellets to the hot surface of the mould. A polymerisation activator is then injected directly into the mould through an atomiser/injector 2 fitted with a Nitrogen purge. The activator is efficiently dispersed into the molten lactam-12 and in-mould polymerisation is initiated. The mould is preferably rotated around one or two axes on introduction of the activator to efficiently dissolve or disperse the activator in the melt. The activator may be introduced using a piston driven pneumatic actuator or a dosing pump with a separate reservoir mounted on the moulding machine. This facilitates controlled delivery of activator, on demand.

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An atomiser head forms a fine mist of activator droplets for rapid mixing. The temperature of the mould is increased to about 200°C over a period of about 10 minutes to complete the polymerisation process. Thus, the material evenly adheres to the mould forming a moulded article 3 of desired thickness, especially thin walled articles.

The lactam and activator are easily handled. The reactive rotational moulding technique is more easily controlled, is faster, can produce a wider range of moulded articles, especially thin walled articles, and lends itself to automation leading to at least semi-continuous operation.

An inert atmosphere, for example provided by nitrogen, is provided in the cavity of the mould to exclude moisture. Re-fillable supply cylinders may be mounted to the mould by a system of valves and regulators to control the flow of gas into the mould.

The various stages in this process are as follows.

Stage 1 – As illustrated in Fig. 1.

A pre-weighed amount of lauralactam pellets (the monomer form of Nylon-12) is placed within the mould 1 e.g. 1.25 kg.

A pre-weighed amount of liquid activator is dispensed into a syringe container and placed in the mounting fixture on the mould. The amount is typically 1.5% by weight of the pre-defined weight of lauralactam pellets being processed e.g. 18.75 ml for 1.25 kg of lauralactam.

The mould breather device, encompassing nitrogen flush tube activator dispensing needle, atomiser head and breather exhaust is inserted into the mould. N₂ flush is

- 8 -

provided for the activator dispensing system to maintain an N₂ atmosphere in the mould and to purge all the activator from the dispensing system after dispensing.

The mould is closed to form a water-tight seal between both halves.

5

Stage 2 – As illustrated in Fig. 2.

A nitrogen flush maintains a slight positive pressure (0.05 bar) within the mould of nitrogen gas. This is to (a) purge any air/moisture within the mould which could adversely affect polymerisation, (b) keep the atomiser head free of any condensed lactam material and, c) to clean atomiser head after injection of activator.

10

The heating cycle commences which heats the mould using embedded resistance heating wires. The mould heats to a typical set point temperature of 170°C (internal face temperature) in 20 minutes.

15

As the mould heats, the lactam pellets also heat up and begin to melt. A large surface area of the mould improves heat transfer and reduces the time required for complete melting of the pellets. Rotation of the mould during pre-heating also ensures uniform and efficient melting of the pellets.

20

A slow rotation speed of typically 3 rpm on the major axis, 3 rpm on the minor axis is used to distribute the pellets on the inside of the mould as illustrated in Fig. 2.

25

Stage 3 – As illustrated in Fig. 3.

After about 20 minutes, the temperature stabilises at 170°C. The lactam pellets have fully melted out to form a liquid with a viscosity similar to that of water.

30

- 9 -

The rotomoulding machine comes to a halt in the vertical plane. The rotation speed in the horizontal is increased to promote shearing motion of the liquid, which now forms a pool on the bottom of the mould.

5 After sufficient time to ensure the liquid pool has become sufficiently agitated, a pneumatic ram is triggered which forces the liquid activator out through a syringe, through the dispensing needle and atomiser head and into the mould. The force applied is regulated by the speed of the ram pushing the syringe. The atomiser head causes the activator to disperse into a cone of minute droplets which drop
10 over the surface of the liquid pool. The combination of the fine activator droplets and the motion of the liquid mean that intimate mixing occurs almost immediately and does not give the activator time to coalesce on the surface of the mould.

15 Typically the injection time is in the order of 10-15 seconds.

The nitrogen flush is used to clean the dispensing needle and atomiser head of any remaining liquid activator.

20 Stage 4 – As illustrated in Fig. 4.

Biaxial rotation re-commences after the activator is injected at a speed of typically greater than 5 rpm on the major and minor axes. This is to create the conditions for the phenomenon of solid body rotation of the molten mass. As the viscosity
25 increases on polymerisation, the speed of rotation may be reduced to maintain solid body rotation and to reduce centrifugal effects and uneven thickness build up.

At the same time, the mould set point temperature is increased to 205°C (typical
30 value, internal face temperature). This takes approximately 5-7 minutes to achieve. The higher temperature is required to reduce the time for polymerisation.

- 10 -

After a sufficient period of time (typically 5-7 minutes to reach temperature, 10 minutes at temperature), the rotation speeds of both axes are reduced by a factor of 50% (typically). This is to account for the rapidly increasing viscosity of the nylon-12 material within the mould. After another 5 minutes, the heaters are
5 turned off and forced air convection cooling commences.

Stage 5 – As illustrated in Fig. 5.

10 Cooling takes approximately 15 minutes to reduce the temperature from 205°C to part removal temperature (typically 80°C).

During this time, it is possible to close off the mould vent and increase the internal pressure to 0.2 bar to reduce warpage.

15

The mould can then be opened, the breather device withdrawn and the moulded nylon-12 part removed.

Referring to Figs 6 to 11 there is illustrated another method of rotomoulding according to the invention. Granules 10 of a thermoplastic material such as the lactam material described above are in this case mixed with a polymerisation
20 activator 11 in a premix vessel 12. The vessel 12 is a sealed vessel with a suitable mixer 13 for mixing the activator 11 and thermoplastics 10 under an inert atmosphere. The vessel 12 has a bottom outlet closed by a valve 15, in this case a flap valve. After premixing, the premix thus formed is loaded into containers
25 such as a bag 16 (Fig 7) under an inert atmosphere. The bag of premixed material is sealed and may be stored until required.

When it is desired to mould a product using the premix a mould 20 is opened and
30 under an inert atmosphere and at room temperature the premix is introduced into the mould 20. If desired, the premix is rotated biaxially in the mould (Fig. 9) and

- 11 -

the temperature of the mould 20 is increased to polymerise the thermoplastic mixture. Typically the temperature is increased by heating the mould walls by imbedded resistance wires to achieve about 205°C on the internal face of the mould. This takes about 10 minutes.

5

At the same time biaxial rotation continues at a speed of typically greater than 5 rpm on the major and minor axes. This is to create the conditions for the phenomenon of solid body rotation of the molten mass. As the viscosity increases on polymerisation, the speed of rotation may be reduced to maintain solid body rotation and to reduce centrifugal effects and uneven thickness build up.

10

After a sufficient period of time, typically about 10 minutes, the rotation speeds of both axes are reduced by a factor of 50% (typically). This is to account for the rapidly increasing viscosity of the nylon-12 material within the mould. After another 5 minutes, the heaters are turned off and forced air convection cooling commences.

15

Cooling takes approximately 15 minutes to reduce the temperature from 205°C to part removal temperature (typically 80°C).

20

During this time, it is possible to close off the mould vent and increase the internal pressure to 0.2 bar to reduce warpage.

The mould 20 can then be opened, the breather device withdrawn and a moulded part 21 removed.

25

The process described with reference to Figs 6 to 11 is particularly advantageous as the premix is formed before being introduced into the mould. The introduction into the mould can be carried out at ambient conditions. The premix is readily prepared and can be stored under an inert atmosphere.

30

The invention provides an integrated process and apparatus for at least semi-continuous rotomoulding. Multiple moulds may be provided on a single machine. If desired, a number of separate moulds may be arranged at separate stations for delivery of pre-polymer material. The stations may, for example, be arranged around a central dispensing unit or may be arranged in-line. A control system may be operated to deliver the pre-polymer material on demand by the individual rotomoulding stations.

10

Because of the low viscosity of the material initial rotation speeds may be relatively high to ensure that a skin of uniform thickness is formed in the mould cavity before the viscosity increases, on polymerisation. This has the advantage that potentially thin walled products may be readily rotationally moulded. In addition, the moulding machine technology used allows the injection of other material into the mould cavity after the outer skin has formed. Thus, for example, polyethylene foam may be injected into the mould cavity.

15

In addition, the low viscosity of the lactam facilitates the forming of very fine details, such as screw threads and the like.

20

Internal pressurisation and/or forced circulation of cooled air/ N₂ to improve cooling efficiency is preferably maintained during cooling to reduce adverse warpage and shrinkage effects.

25

Typically a batch of 1kg lactam is placed in a container and a vacuum is applied. An inert atmosphere is introduced by nitrogen. The container is opened while maintaining the inert atmosphere and an amount of about 15g of an activator which is typically in a liquid form is added. The container is closed and the contents are mechanically agitated to disperse the activator in the lactam. The premix thus formed can be stored indefinitely in an inert atmosphere.

30

In moulding, the prepared premix is dispersed into the mould, air is purged from the mould and the moulding process is carried out as described above.

5 Optionally the premix may include a filler which may be present in an amount of from 10 to 70% by volume. The filler may be of a ceramic material, glass, talc or any suitable inert material. It may be in the form of particulates, microbeads, microspheres or fibres. For processing, typically the filler is added to the premix under an inert atmosphere.

10

The advantages of using such fillers are lower costs, enhanced properties and improved processing. The fillers increase viscosity and reduce flow instability on flow allowing for more even wall thicknesses.

15 Fillers in formulations for rotational moulding can be accommodated because, in addition to the premixing described above the rotational moulding process mixes and agitates the filler during processing to ensure homogenous distribution of the fillers in the final product. The use of a large hollow cavity facilitates the placement of materials with a relatively large bulk density into the mould to
20 produce higher density material than with more conventional moulding where the entire cavity is filled with the material. Using filler materials generally involves increasing the processing temperatures by typically 10°C, depending on the type of and amount of filler used.

25 It will be appreciated that the process and apparatus may be applied to the rotational moulding of any suitable thermoplastic material. In one case the material is a lactam-based prepolymer. Such a prepolymer may be laurylactam (Nylon 12), caprolactam (Nylon 6) or a blend of both at different ratios dependent on the final properties desired. In some cases a lower viscosity melt is obtained
30 using such blends which is generally preferable if there are added fillers.

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The thermoplastic may also include or comprise one or more of PBT, ABS, acrylic, polycarbonate, lactam, mixture, blends or copolymers thereof.

- 5 The invention is not limited to the embodiments hereinbefore described which may be varied in construction and detail.

CLAIMS

1. A method of rotomoulding comprising the step of forming a reactant thermoplastics melt in the mould.
- 5 2. A method as claimed in claim 1 wherein the reactant thermoplastics melt comprises a mixture of a thermoplastic pre-polymer and a polymerisation activator.
- 10 3. A method as claimed in claim 2 wherein the thermoplastics pre-polymer and the polymerisation activator are mixed in the mould.
4. A method as claimed in claim 2 where in the thermoplastics pre-polymer and the polymerisation activator are mixed prior to delivery into a mould.
- 15 5. A method as claimed in claim 3 or 4 wherein the mixing is carried out under an inert atmosphere.
6. A method as claimed in claim 5 wherein the inert atmosphere is a nitrogen atmosphere.
- 20 7. A method as claimed in any of claims 4 to 6 wherein the pre-polymer and polymerisation activator are mixed to form a pre-mix which is stored in a holding container prior to delivery into the mould.
- 25 8. A method as claimed in any of claims 4 to 7 wherein the mixture is introduced to the mould at ambient temperature conditions.
9. A method as claimed in any of claims 4 to 8 wherein the mixture is introduced into the mould under an inert atmosphere.
- 30

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10. A method as claimed in any of claims 4 to 9 wherein, after introduction of the mixture, the mould is heated to polymerisation temperature.
- 5 11. A method as claimed in any of claims 4 to 10 wherein, after introduction of the mixture, the mould is rotated.
12. A method as claimed in claim 11 wherein the mould is rotated biaxially.
- 10 13. A method as claimed in claim 12 wherein the biaxial rotation is at a speed of greater than 5 r.p.m.
14. A method as claimed in claim 3 comprising the steps of: -
- 15 delivering a thermoplastics pre-polymer into the mould; and
- introducing a polymerisation activator to the thermoplastic pre-polymer in the mould.
- 20 15. A method as claimed in claim 14 wherein the polymerisation activator is introduced into the mould separately from the pre-polymer.
16. A method as claimed in claim 15 wherein the polymerisation activator is injected into the mould to form a reactant melt in the mould.
- 25 17. A method as claimed in any of claims 14 to 16 including the steps of:
- placing the thermoplastic pre-polymer in a mould;
- heating the mould;
- 30 injecting an activator into the pre-heated material in the mould; and

- 17 -

further heating the mould.

18. A method as claimed in claim 17 comprising the steps of: -

5

placing thermoplastic pre-polymer in a mould;

heating the mould;

10

rotating the mould;

injecting an activator into the preheated material in the mould; and

further rotation and heating of the mould.

15

19. A method as claimed in claim 17 or 18 wherein the mould is pre-heated to a temperature of from ambient temperature to 170°C.

20. A method as claimed in claim 19 wherein the mould is pre-heated to a temperature of approximately 170°C.

20

21. A method as claimed in any of claims 17 to 20 wherein the mould, after injection of the activator, is further heated to a temperature of from 170°C to 205°C over a period of from 5 to 10 minutes.

25

22. A method as claimed in claim 21 wherein the mould is further heated to a temperature of about 200°C over a period of about 10 minutes to ensure complete polymerisation.

30

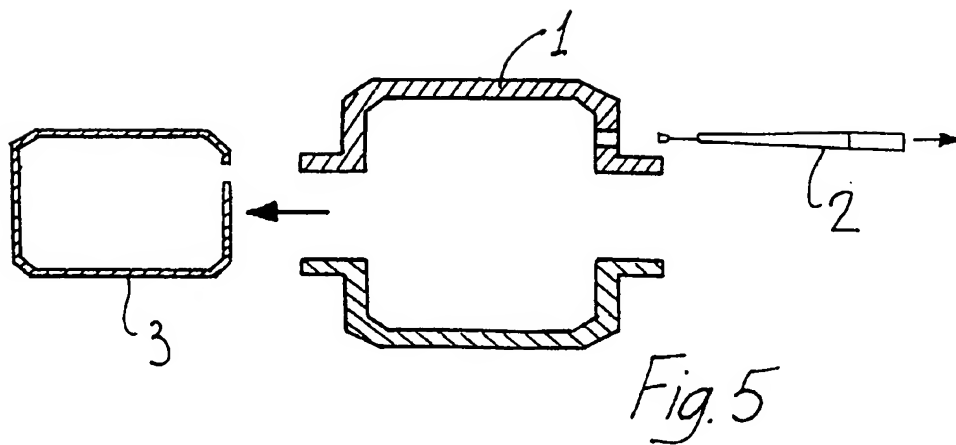
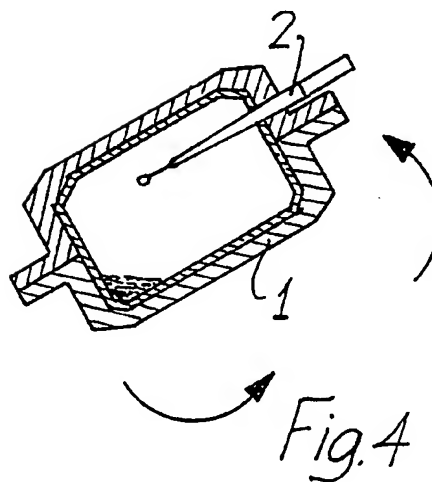
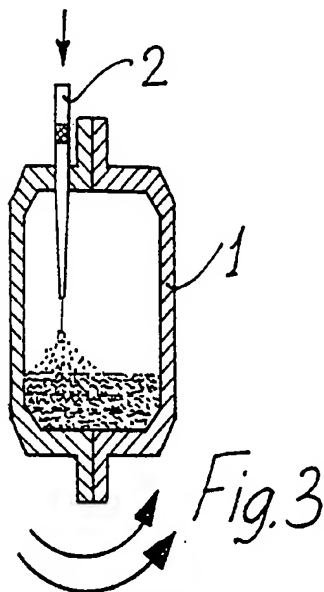
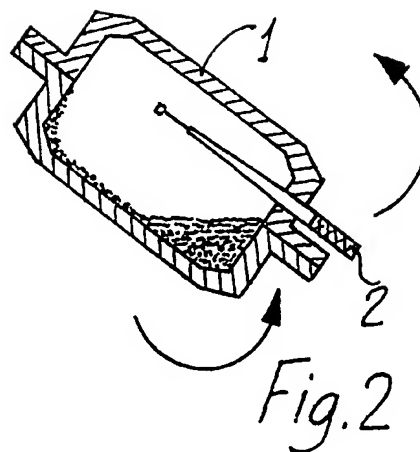
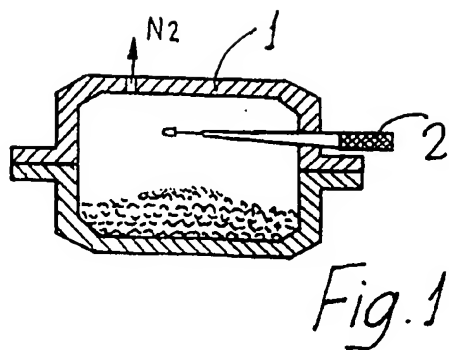
23. A method as claimed in any of claims 16 to 22 including the step of purging the activator line after injection of the activator.

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24. A method as claimed in any of claims 19 to 23 including the step of rotating the mould during preheating.
- 5 25. A method as claimed in any of claims 16 to 24 including the step of rotating the mould after injection of the activator to dissolve or disperse the activator in the preheated melt.
26. A method as claimed in an of claims 16 to 25 including the step of rotating
10 the mould biaxially after dispersion of the activator in the melt.
27. A method as claimed in claim 26 wherein the biaxial rotation is at a speed of greater than 5 rpm.
- 15 28. A method as claimed in any preceding claim wherein the thermoplastic is selected from one or more of PBT, ABS, acrylic, polycarbonate, lactam, mixtures, blends or copolymers thereof.
29. A method as claimed in any preceding claim wherein the thermoplastic is a
20 lactam material.
30. A method as claimed in claim 29 wherein the activator is an activator for producing an ionically polymerised lactam.
- 25 31. A method as claimed in claim 29 or 30 wherein the lactam is lactam-12.
32. A method as claimed in any preceding wherein the thermoplastic includes a filler material.
- 30 33. A method of rotomoulding substantially as hereinbefore described.

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34. A rotomoulding apparatus including a mould having an injector means for injecting an activator into the mould for in-mould reactive moulding.
- 5
35. Apparatus as claimed in claim 34 wherein the mould has integral mould heating and cooling means.
36. A rotomoulding apparatus as claimed in claim 34 or 35 including a flush means for flushing the injector after injection of the activator into the mould.
- 10
37. A rotomoulding apparatus substantially as hereinbefore described with reference to the accompanying drawings.
- 15
38. An article whenever rotationally moulded by a method as claimed in any of claims 1 to 33 and /or using an apparatus as claimed in any of claims 34 to 37.



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